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A REAGENT FOR THE SIMULTANEOUS MICROSCOPIC DETERMINATION OF QUARTZ AND HALIDES

By
Henry Nusbaum

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WHITE SANDS MISSILE RANGE, NEW MEXICO

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ABSTRACT

A reagent has been discovered whereby the techniques of dispersion staining microscopy and spot testing may be combined to distinguish halite from quartz in samples of atmospheric dust. The preparation of the reagent and its application are discussed.

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INTRODUCTION

In a previous study of the mineral composition of dust in the atmospheric aerosol over White Sands Missile Range (WSMR), New Mexico (Hoidale et al., 1966), quartz was one of several minerals color identified by dispersion staining microscopy. With this method, halite could have been identified as quartz because halite is an optically isotropic mineral having the same refractive index (1.544) as the omega index of quartz. If significant quantities of halite are present in the local dust samples, the differentiation between quartz and halite becomes important in dispersion staining assays of quartz concentration (Hoidale et al., 1966).

In the earlier study (Hoidale et al., 1966) quartz was identified using ethynylbenzene as the immersion fluid. The quartz was distinguished from halite through use of polarized light, but it became obvious that as the particle diameter reached a lower limit of four to five microns, differentiation between the two minerals by dispersion staining microscopy became difficult. Therefore, an immersion fluid was sought which would unequivocally identify quartz to the exclusion of halite. A technique evolved which combines spot testing with dispersion staining whereby a single reagent may be used to eliminate halite from the quartz count by imparting to the soluble halides a characteristic "cocklebur" appearance. Also, this reagent permits a combined count of these halides and an indirect determination of the amount of halite present.

This report discusses the preparation of the reagent and the method of application to be followed in processing a dust sample.

REAGENT COMPONENTS

The following factors are necessary for the identification of quartz by dispersion staining microscopy:

- a) An immersion fluid having the same refractive index at the sodium D line (n_D , $D = 589.3 \text{ m}\mu$) as quartz (1.544) and

a larger dispersion ($n_F - n_C$), where $F = 486.1 \text{ m}\mu$ and $C = 656.3 \text{ m}\mu$.

b) An absence of other minerals having a refractive index equal to that of quartz.

The reagent having the desired refractive index, dispersion, and stability is a solution of ethyl salicylate, phenol, 1-bromonaphthalene, and mercurous perchlorate. This solution will be referred to here as the "4BR reagent". The function of each component is:

ETHYL SALICYLATE: This liquid ($n_D = 1.5251^{20}$) is the main solvent. It is used to dissolve the solid phenol and bring the original refractive index slightly below 1.54.

PHENOL: In an ethyl salicylate solution, the polar molecules of phenol ($n_D = 1.5509^{21}$) are able to dissolve mercurous perchlorate, $\text{Hg}_2(\text{ClO}_4)_2$, but not the halides. The small amount of water absorbed by the phenol from the air probably dampens the surface of the halite particles; this dampening may be necessary for the spot test reaction. This, however, has not been proved.

1-BROMONAPHTHALENE: 1-bromonaphthalene ($n_D = 1.6588^{19}$) is added to adjust the refractive index to 1.544.

MERCUROUS PERCHLORATE: The mercurous ion, Hg_2^{++} , reacts with the halide ions to form insoluble mercurous halides. The mercurous reagent was chosen because it is more stable than other insoluble halide-forming ions. The perchlorate salt was selected for its high solubility (368 g/100 cm³ water at 20° C) and good stability.

4BR PREPARATION

The 4BR reagent was prepared by the following method.

Approximately 3 ml (2g) of phenol crystals were poured into a test tube, then enough ethyl salicylate was added to cover 3/4 of these. When the phenol was nearly dissolved, about 10 mg of dried mercurous perchlorate was added.

The addition of mercurous perchlorate (prepared by the method described later) caused the phenol in the solution to turn a medium yellow. This may have been caused by the small trace of mercuric ion that is always present in mercurous compounds.

After the mercurous perchlorate had nearly dissolved, a few drops of 1-bromonaphthalene were added. This appeared to stop the color-forming reaction. After the last of the phenol had dissolved, enough 1-bromonaphthalene was added to adjust the refractive index to 1.544 as read on a Bausch and Lomb "Abbe-3L" Refractometer. From readings obtained on this instrument, the dispersion was calculated to be 0.028 at 25° C.

4BR STABILITY

This reagent, stored in a tightly stoppered green dropping bottle, retained its original optical and chemical properties for more than a month. Various factors influenced this stability, but the decomposition mechanisms were not investigated. The deterioration of the reagent was indicated in all cases by loss of the yellow color and the formation of a gray precipitate on the bottom of the bottle.

Exposure to light and air caused the reagent to decompose in a few hours. It was apparent that the $\text{Hg}_2(\text{ClO}_4)_2$ must be free of the mercuric and carbonate impurities which could result from its exposure to air.

Solutions containing 75 percent by weight or more phenol were found to have the best stability and to produce the best spot tests.

4BR APPLICATION

For the microscopic determination of quartz, a small amount of the dust sample is placed on a slide and covered with a drop of 4BR with a cover glass placed over it. The slide is then allowed to "develop" for 20 minutes. Very little if any change was noticed on slides "developed" from approximately 20 minutes to 20 hours.

After the 20 minutes, the quartz exhibited the characteristic sky-blue color with a red-orange halo when viewed with a positive phase contrast microscope. Halite and the other halides appear as light brown "cockleburs" - a central particle from which dozens of long brown "whiskers" or "needles" are growing. Soluble sulfates take on the appearance of a central particle surrounded by black "bushes" of Hg_2SO_4 . Insoluble halides, sulfates, and other minerals produce no reaction.

It was hoped that by dilution of one or more of the reagent's components, very few whiskers would grow, leaving the halite as blue particles with a few protruding needles. Unfortunately, all attempts to reduce the phenol concentration resulted in an unstable solution, while reducing the mercurous perchlorate concentration caused the needles to break loose from the particles.

Halite concentration can be indirectly determined by subtraction if it is assumed that the mineral concentrations are uniform for the total individual sample collected. This is accomplished by counting one slide, as previously described, then counting a second slide using ethynylbenzene as the immersion fluid. With ethynylbenzene, both quartz and halite appear sky-blue with a red-orange halo. The number of counts from the ethynylbenzene slide minus the quartz count determined with the 4BR reagent would, therefore, be the halite count.

PREPARATION OF MERCUROUS PERCHLORATE

Since mercurous perchlorate is not commercially available, an electrolytic preparation was used.

In subdued light, approximately 30 ml of mercury and 60 ml of .5M Na_2CO_3 were placed in a 100 ml beaker. Teflon insulated wire was run through the solution to the bottom of the mercury (anode) and a stainless steel cathode placed in the sodium carbonate. A potential of 1.9 volts was applied for five hours while the cell was being agitated on an oscillating plate. This agitation was continued for thirty minutes after the current was turned off to let the mercury reduce any mercuric ions which may have been formed.

Next, the cathode was removed and the carbonate solution pipetted off. The yellow-brown mercurous carbonate and a small amount of mercury were scooped out and washed with two 50 ml portions of distilled water. Then, most of the mercurous carbonate was titrated with .6N perchloric acid to form a mercurous perchlorate solution. The excess carbonate eliminated the possibility of having perchloric acid in the product, while the mercury again reduced any mercuric ion formed.

The mercurous perchlorate solution, withdrawn with a pipette, was boiled with a Teflon boiling chip and dried at 90° C in a vacuum oven. The solid $\text{Hg}_2(\text{ClO}_4)_2$ was stored in a tightly capped green bottle. For long storage periods, it is probably best to flush the bottle and crystals with a dry, inert gas before sealing to prevent oxidation and reaction with atmospheric carbon dioxide.

The product was dissolved in distilled water and precipitated with sodium chloride solution to form calomel for quality checking. Sodium carbonate solution was added to the remaining liquid as a test for mercuric ion. No red-brown precipitate (mercuric carbonate) was formed. An additional check for the purity of the mercurous perchlorate crystals was made by running an infrared absorption spectrum on a Perkin-Elmer 521 infrared spectrophotometer using the potassium bromide-pellet technique. There was no indication of decomposition of the perchlorate or presence of carbonate or bicarbonate ions.

POSSIBILITIES FOR FURTHER RESEARCH

It is hoped that the technique may be expanded to include the identification of the various halide and sulfate condensation nuclei, since the "whiskers" formed by the 4BR reagent are many times the radius of the particle. Possibly the addition of an absorption indicator to the 4BR reagent will give distinctive appearances to the whiskers formed on the halide and sulfate nuclei. Fluorescein, Congo red, bromophenol-blue, and 3', 3'', 5', 5''-tetrabromophenolsulfonaphthalein were tried but did not accomplish this. Perhaps raising the pH of the 4BR reagent, possibly with sodium or potassium phenoxide, would have permitted them to differentiate the individual halides, but time did not permit this experiment.

CONCLUSION

The use of 4BR improves the accuracy and ease of counting quartz particles in samples of atmospheric dust by excluding halite from the count. The reagent obviates polarized light and rotation of the microscope stage in distinguishing halite from quartz. The differentiation of these two minerals by dispersion staining microscopy is a problem with the smaller particles.

The simultaneous determination of submicron particles of quartz, halite, other soluble chlorides, soluble fluorides, soluble bromides, soluble iodides, and soluble sulfates may be possible by modifying the composition of the reagent. Such a modification will require more extensive research.

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